

**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

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IN RE APPLICATION OF:

Kazuhiro YOSHINO et al.

Application No.: 10/560,239

Confirmation No.: 1480

Filed: December 12, 2005

Art Unit: 3761

For: ABSORBING MATERIAL AND  
ABSORPTIVE ARTICLE USING  
THE SAME

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EXAMINER: WIEST, Philip R.

DECLARATION UNDER 37 C.F.R. 1.132

COMMISSIONER FOR PATENTS

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Madam:

I, Tomoki KAWAKITA, residing in Hyogo-ken, Japan, hereby declare  
and state as follows:

1. I am one of the co-inventors of U.S. Application Serial No.  
10/560,239 filed on December 12, 2005, entitled ABSORBING MATERIAL  
AND ABSORPTIVE ARTICLE USING THE SAME, its prosecution before the  
United States Patent and Trademark Office and the references cited therein.
2. I am a graduate from Nagoya Institute of Technology, faculty of  
applied chemistry in the year 1991, majoring in applied chemistry.
3. I have been employed in Sumitomo Seika Chemicals Co., Ltd. in  
the year 1991 and have been assigned to the Research Laboratories.

4. I have been involved in the research and development of water-absorbent resin in the years 2002-2003 and since the year 2007.

5. The following experiments were conducted by myself or under my direct supervision and control in order to verify that the absorbent article of the present invention is distinguishable from those of Wada (U.S. Patent No. 6,150,582). Specifically, an absorbent article was produced using a water-absorbent resin obtained by a follow-up example of Wada prepared in the previous declaration submitted on June 12, 2008, and the properties of this absorbent article and the absorbent article of the present invention were compared.

## EXPERIMENTAL METHOD

### FOLLOW-UP EXPERIMENT OF WADA

#### 1. REFERENTIAL EXAMPLE 4 (Corresponding to Water-absorbent resin (4)) OF WADA

The properties of Water-absorbent resin (4) obtained by a follow-up example of REFERENTIAL EXAMPLE 4 of Wada were evaluated in the following manner.

##### 1-1. Synthesis of Water-Absorbent Resin (Unless specified otherwise, half the scale of that disclosed in REFERENTIAL EXAMPLE 4 of Wada was used.)

A reaction solution was prepared by dissolving 3.235 g of polyethylene glycol diacrylate (average molar number of added ethylene oxide: 8) into 2,750 g

of an aqueous solution of sodium acrylate with a neutralization ratio of 65 mol % (monomer concentration: 30 wt %). Next, this solution was degassed under a nitrogen gas atmosphere for 30 minutes, and then supplied into a reaction vessel as prepared by capping a stainless-steel-made double-arm type kneader of a capacity of 5 liters having two sigma type wings and a jacket. While maintaining the reaction solution at 30°C., the atmosphere inside the system was replaced with a nitrogen gas. Next, while the reaction solution was stirred, 0.955 g of 2,2'-azobis(2-amidinopropane) dihydrochloride, 0.48 g of sodium persulfate and 0.05 g of L-ascorbic acid were added, so that a polymerization reaction got started about 1 minute after. The polymerization was carried out at 30° to 80°C, and the resultant hydrogel polymer was separated out 60 minutes after the initiation of the polymerization.

The resultant hydrogel polymer had a finely divided diameter of about 5 mm. This finely divided hydrogel polymer was spread on a 50-mesh wire net and dried at 150°C. with hot air for 90 minutes. Then, the resultant dried product was pulverized with a ball-mill and further classified with a wire net of 20 mesh, thus obtaining a formless pulverized water-absorbent resin precursor (A).

A surface-crosslinking agent comprising 0.3 g (1 part) of propylene glycol, 0.0075 g (0.025 parts) of ethylene glycol diglycidyl ether, 0.6 g (2 parts) of water, and 0.3 g (1 part) of isopropyl alcohol was mixed with 30 g (100 parts) of the resultant water-absorbent resin precursor (A). The mixture was mixed in a 1-L separable flask by spraying, while stirring with agitation blades. The resultant mixture was heated at 185°C. for 45 minutes, thus obtaining a water-absorbent resin (B), of which the average particle diameter was 420  $\mu\text{m}$ .

1-2. Water-Retaining Capacity of Physiological Saline (The Present Invention)

Two grams of a water-absorbent resin was placed in a cotton bag (Cottonbroad No. 60, width 100 mm × length 200 mm), and the cotton bag was placed in a 500 mL-beaker. Physiological saline was poured into the cotton bag in an amount of 500 g at a time, and the saline was dispersed so as not to generate a lump of the water-absorbent resin. The upper part of the cotton bag was tied up with a rubber band, and the cotton bag was allowed to stand for 1 hour, to sufficiently swell the water-absorbent resin. The cotton bag was spin-dried for 1 minute with a spin dryer (manufactured by Kokusan Enshinki Co., Ltd., H-122) set to have a centrifugal force of 167G, and a weight  $W_c$  (g) of the cotton bag containing swollen gels after the dehydration was determined. The same procedures were carried out without adding a water-absorbent resin, and an empty weight  $W_d$  (g) of the cotton bag upon wetting was determined. The water-retaining capacity was calculated in accordance with the following formula:

$$[\text{Water-Retaining Capacity of Physiological Saline (g/g)}] = (W_c - W_d)/2$$

1-3. Water Absorption of Physiological Saline (The Present Invention)

In a 1000 mL beaker, 2 g of a water-absorbent resin was dispersed in 1000 g of physiological saline (0.9% by weight aqueous sodium chloride), and the dispersion was gently stirred for 1 hour to sufficiently swell the resin. On the other hand, the physiological saline containing the swollen gel was filtered with a JIS standard sieve having an opening of 75  $\mu\text{m}$ , the JIS standard sieve of which weight  $W_a$  (g) was previously determined. The filtered sieve was allowed to stand for 30 minutes in a state so that the sieve was tilted at a tilt angle of about

30 degrees to the horizontal to remove excess physiological saline from the water-absorbent resin. After a weight  $W_b$  (g) of the sieve containing the swollen gel was determined, the water absorption was calculated in accordance with the following formula:

$$[\text{Water Absorption of Physiological Saline (g/g)}] = (W_b - W_a)/2$$

1-4. Production of Absorbent Article (Water-absorbent resin (4) of WADA)

A mixture prepared by dry-blending 6 g of the water-absorbent resin (B) obtained in Preparation 1-1 (in other words, a follow-up sample of Water-absorbent resin (4) of WADA) and 10 g of pulverized pulp with a mixer was sprayed on tissue paper having a size of 40 cm  $\times$  10 cm and a weight of 1 g. Thereafter, tissue paper having the same size and the same weight as above was layered over the top, and formed into a sheet. A 196 kPa load was applied to the entire sheet for 30 seconds to press the sheet, thereby giving an absorbent having a concentration of a water-absorbent resin of 37.5% by weight. The resulting absorbent was interposed between a polyethylene air-through, porous liquid-permeable sheet having a size of 40 cm  $\times$  12 cm and a basis weight of 20 g/cm<sup>2</sup> and a polyethylene impermeable sheet having the same size and the same basis weight as above to give an absorbent article using the absorbent having a concentration of a water-absorbent resin of 37.5% by weight. Similarly, 10 g of the water-absorbent resin (B) and 10 g of pulverized pulp were used to give an absorbent having a concentration of a water-absorbent resin of 50% by weight and an absorbent article using the absorbent.

1-5. Measurement methods of Absorbent Article

## i) Preparation of Artificial Urine

Sixty grams of sodium chloride, 1.8 g of calcium chloride dihydrate, 3.6 g of magnesium chloride hexahydrate, and a proper amount of distilled water were placed in a 10 L container, and were completely dissolved. Next, 0.02 g of polyoxyethylene nonyl phenyl ether was added to the mixture, and distilled water was further added thereto to make up a weight of 6000 g of the entire aqueous solution. Further, the aqueous solution was colored with a small amount of Blue No. 1, to give an artificial urine.

## ii) Permeation Time

A cylinder having an inner diameter of 3 cm was placed near the center of the absorbent article, and 50 mL of the artificial urine which was previously kept at  $25^{\circ} \pm 0.2^{\circ}\text{C}$  in a thermostatic water tank was poured therein. At the same time, a stopwatch was clicked to start to determine the time required until the artificial urine was completely permeated into the absorbent article (first time). Next, the above-mentioned cylinder was removed and the absorbent article was kept in that state. After 30 minutes passed from the beginning of the first pouring of the artificial urine, the above-mentioned cylinder was again placed on the same position as before, and 50 mL of the artificial urine was poured therein. At the same time, the stopwatch was clicked to start to determine the time required until the artificial urine was completely permeated in the absorbent article (second time). Further, the permeation time was determined up to three times for an absorbent article using the absorbent having a water-absorbent resin concentration of 37.5% by weight, and five times for an absorbent article using

the absorbent having a water-absorbent resin concentration of 50% by weight in the same manner as above.

iii) Amount of Re-Wet

After 60 minutes passed from the final determination of the above-mentioned permeation time, filter papers (Toyo Roshi Kaisha, Ltd., No. 2), which was cut into pieces having a size of 10 cm  $\times$  10 cm, were stacked in layers to obtain an about 80 g portion of filter papers, and its dry weight (g) was determined. The filter paper was placed on the central part of the absorbent article, and a 5 kg weight (base area=10 cm  $\times$  10 cm) was placed thereon to apply a load for 5 minutes. Thereafter, the weight was removed therefrom and the weight (g) of the filter paper absorbing the re-wet liquid was determined. The amount of re-wet was calculated by subtracting the weight (g) of the dry filter paper from the weight (g) of the filter paper absorbing the re-wet liquid.

iv) Diffusion Length

The diffusion size (cm) in the longitudinal direction of each absorbent article in which the artificial urine was permeated was determined within 5 minutes after the determination of the above-mentioned amount of re-wet. Here, the numerical values of decimal places were rounded to the nearest whole number.

## 2. MEASUREMENT RESULTS

### 2-1. Measurements of Properties of Water-absorbent resin (B)

Water Absorption and Water Retaining Capacity for Water-absorbent resin (B) (a follow-up example of Water-absorbent resin (4) of Wada) were measured. The results are shown in Table I. Further, for the sake of even fairer comparison, the water-absorbent resins having the two lowest water retaining capacity among the water-absorbent resins used in Examples and Comparative Examples of the present invention, in other words, the water-absorbent resins of Preparation Example 5 and Preparation Example 11 were chosen as comparative subjects in the present experiments. The data for these water-absorbent resins are together shown in Table I.

**Table I Properties of Water-Absorbent Resins Evaluated by Absorbent Article**

Ex. No.	Water absorbent resin used	Water Absorption (g/g)	Water Retaining Capacity (g/g)
Example 3 of Present Specification	Preparation Example 5	72	52
Comparative Example 3 of Present Specification	Preparation Example 11	50	30
	Referential Example 4 of WADA	46	29

The properties of Water-absorbent resin (B) were poorer than the water absorption properties shown in Wada, as shown in the previous Declaration filed June 12, 2008.



**Table II      Comparison Between the Properties of Water-absorbent resin (B) and the Properties Disclosed in Wada**

	Parameters of WADA					Parameters of the Present Invention	
	Absorption Capacity Under No Load	Absorption Capacity Under Load	Amount of Water-Soluble Component	Absorption Speed	Urine Resistance Index	Water-Retaining Capacity	Water Absorption
Water-Absorbent Resin (B)	38	26	7	54	0	29	46
Values Disclosed in Wada	60	13	13	48	0	-	-

The values shown in Table II were evaluated in the same manner as in the previous declaration dated June 12, 2008. Please refer to the details to the previous declaration.

## 2-2. Measurements Results of Absorbent Article

The properties of Absorbent article obtained by using Water-absorbent resin (B) were measured. The results are shown in the following Tables III and IV, together with the absorbent articles obtained by using the water-absorbent resins in Preparation Example 5 and Preparation Example 11 shown in Table I.

**Table III Absorbent Article Using Absorbent Having Concentration of Water-Absorbent Resin of 37.5% by weight**

Ex. No.	Permeation Time (sec)				Amount of Re-wet (g)	Diffusion Length (cm)
	1st	2nd	3rd	Total		
Example 3 of Present Specification	27	32	40	99	35.0	27
Comparative Example 3 of Present Specification	31	25	40	96	54.7	30
Absorbent Article from Water-absorbent resin (B)	21	14	21	56	40.1	27

**Table IV Absorbent Article Using Absorbent Having Concentration of Water-Absorbent Resin of 50% by weight**

Ex. No.	Permeation Time (sec)						Amount of Re-wet (g)	Diffusion Length (cm)
	1st	2nd	3rd	4th	5th	Total		
Example 3 of Present Specification	24	22	25	28	32	131	22.9	21
Comparative Example 3 of Present Specification	20	18	22	26	37	123	48.2	24
Absorbent Article from Water-absorbent resin (B)	20	13	17	26	30	106	50.9	31

## DISCUSSION

As shown in Table I, the Water-absorbent resin concretely described in Wada cannot satisfy both the Water Absorption (a) and the Water-Retaining Capacity (b), which are the required elements of claim 1 of the present invention.

Further, it is verified from Tables III and IV that the properties of the Absorbent article obtained by using Water-absorbent resin (B) are poorer than the properties of the Absorbent article of Examples of the present application, especially in the amount of re-wet. The results support that the properties of the

Absorbent article are influenced by the properties of the Water-absorbent resin used.

Therefore, the Water-absorbent resin disclosed in Wada does not inherently have the properties of the Water-absorbent resin described in the present invention, so that the present invention is a completely different technique from the invention described in Wada. Moreover, it is not obvious to arrive at the constitution of the present invention from Wada that does not disclose nor teach the above required elements or give any descriptions on their effects.

**Statement Under 18 U.S.C. § 1001**

I hereby declare that all statements made herein of my own knowledge are true, and that all statements made on information and belief are believed to be true; and further, that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Dated: July 6, 2009

By Tomoki Kawakita

Tomoki KAWAKITA